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Research Article

APPLICATION OF RAW PEACH STONES FOR HEXAVALENT CHROMIUM REMOVAL FROM AQUEOUS SOLUTION USING COLUMN SYSTEM

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ABSTRACT

Raw biosorbent waste is a cheap and environmentally friendly material that provides good cost-benefit for the industries that use it. The objective of this study is to improve the feasibility of raw local peach stones (RPS) waste for the removal of hexavalent chromium from aqueous solution using column system. The characterization of the adsorbent (RPS) was done by using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The impact of various parameters such as flow rate (1.5,3 and 5 ml/min) and height bed (1,2 and 3 cm) on Cr(VI) adsorption onto RPS were investigated. Two models were proposed to illustrate column breakthrough curve obtained at different flow rates and bed heights. The obtained experimental results showed a better adsorption efficiency at a low flow rate (1.5 ml/min) and a bed height of 3cm. Thus this work provides the high potential of raw peach stones (RPS) for the removal of Cr(VI) ions from aqueous solution.

Keywords: Heavy metal; Biosorbent waste; Adsorption; Breakthrough; Flow rate; Bed height.

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1. INTRODUCTION

The excessive industrial discharges without any appropriate treatment containing heavy metals represents a significant and long-term environmental hazard [1]. These heavy metals and their compounds are potential pollutants that could be particularly problematic due to their mobility, stability and toxicity [2]. Heavy metals have great harm to environment because they accumulate in creatures and plants and enter the human body through food chain [3]. Depending on their speciation and concentration, heavy metals also may cause adverse effects on environmental systems [4]. Chromium is one of the most highly toxic heavy metal generated from many industrial activities, such as mining, leather tanning, textile dyeing, electroplating, metallurgy, wood preservation and petroleum refining [5, 6]. In aqueous solutions, chromium usually exists in the oxidative states of hexavalent chromium Cr (VI) and trivalent chromium Cr(III), and the toxicity of chromium is closely related to the valence state [7]. Hexavalent chromium is 100 times more toxic than trivalent chromium [8]. Cr(VI) compounds are highly toxic, mutagenic and carcinogenic [9]. Therefore, due to its high toxicity it is very necessary to reduce concentration of the Cr(VI) to an acceptable level before discharging it into the environments. In addition, the cost of metallic chromium is significant and it is possible to recover it from the wastewater [10]. Based on its health effect and impact on the environment the maximum permissible limit (MPL) for Cr(VI) in inland and drinking waters are 0.1mg/L and 0.05 mg/L respectively according to the guidelines recommended by the World Health Organization (WHO) [11]. Several treatment technologies have been developed to remove chromium from industrial wastewater, such as chemical precipitation, ion exchange, membrane separation, reduction, electrochemical precipitation, electrodialysis, electrocoagulation, solvent extraction and adsorption [12-14]. However, these methods have technical or economic limits, such as high operational cost, generation of toxic sludge, chemical requirement and incomplete removal [15, 16]. Compared to these methods, the adsorption process has been observed to be the most suitable technology for heavy metal removal from wastewater due to low treatment cost, simple operation, high removal efficiency and availability of a large number of adsorbents. The cost of adsorbents is also an important parameter. Agricultural by-products exist in large amounts, which represent consequently a solid pollutant to the environment. In recent years, special attention has been focused towards valorization of these wastes for their uses in adsorption treatment. These wastes can be used raw or treated (physically or chemically) [17-19]. Fixed-bed columns were widely used in various chemical industries for their operation [20]. The performance of packed beds is described through the concept of the breakthrough curve [20]. In the present study, peach stones were employed as a low cost and efficient adsorbent material for removal of Cr(VI) ions from aqueous solutions in a fixed bed column.

Utilization of the biomaterial without any physical or chemical treatment is important from both environmental and economical viewpoints and is one of the important features of this study.

2. EXPERIMENTAL

2.1. Preparation of biosorbent (RPS)

The biosorbent used in this study was prepared from agricultural waste biomass: peach stones. Firstly, the collected raw peach stones were washed manually several times with tap water to remove the adhering impurities followed by washing with distilled water. After drying in an oven at 110°C for 24h they were crushed and sieved to achieve a fraction with a 0.315-1mm particle size. Finally, the resulting biosorbent obtained without any physical or chemical treatment was stored in a desiccator for the adsorption experiments. Synthesis steps for biosorbent from raw peach stones are shown in Figure 1.

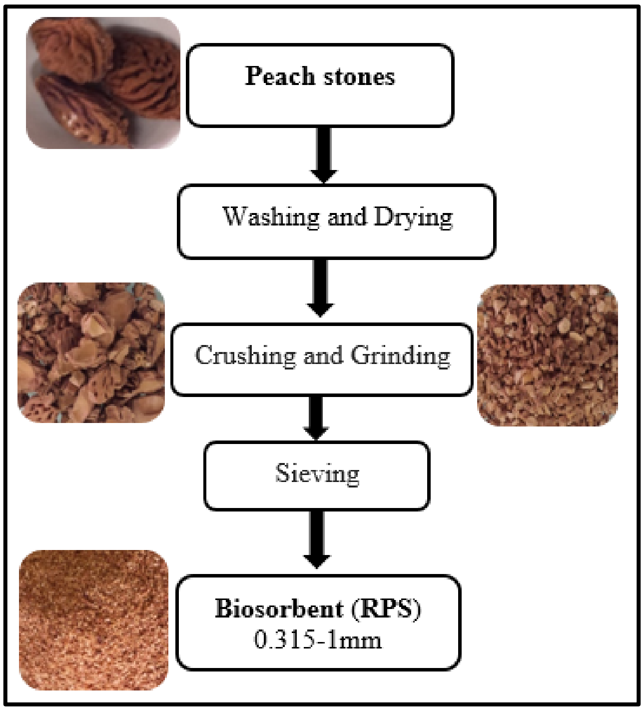

Fig.1. Synthesis steps for biosorbent from raw peach stones

2.2. Characterization of RPS

The characterization of the prepared biosorbent (RPS) is an important factor to explain the mechanism of biosorption process for removal of Cr(VI) from aqueous solutions [21]. Two techniques were used for the biosorbent characterization. The surface morphology of the RPS biosorbent was analyzed by scanning electron microscope (SEM) (Quanta 650). The functional groups at the surface of the biosorbent were determined using Fourier Transform Infrared spectroscopy (Bruker ALPHA) at wavelengths in the range 400–4000 cm^{-1}.

2.3. Preparation of Cr (VI) solution

A synthetic chromium solution was prepared by dissolving potassium dichromate($K_2Cr_2O_7$) in distilled water, after each adsorption experiment, the solution was filtered and the residual concentration of Cr(VI) was determined by using a spectrophotometer (UV-1800 SHIMADZU) at λmax 540 nm after complexing with 1, 5-diphenylcarbazide in acidic medium [22]. The initial pH of working solutions for biosorption experiments was adjusted to the desired value by adding hydrochloric acid HCl (0.1M).

2.4. Fixed-bed column experiments

Fixed bed adsorption used in this study was performed in a glass column with height of 15 cm and internal diameter of 2 cm. The column was packed with the RPS between glass wool and supported by inert glass beads as shown in Figure 2. A solution of Cr(VI) was passed through the column at desired flow rates using a peristaltic pump. The initial concentration of Cr(VI) ions was held constant at 30mg/L at pH2 .Two column parameters affecting biosorption of Cr(VI) ions onto RPS were investigated as follows:

a) Effect of flow rate: Flow rate was varied from 1.5 and 5ml/min with constant bed height of 1cm.

b) Effect of bed height: bed height was varied as 1, 2 and 3cm with constant flow rate of 1.5ml/min.

Subsequently samples were collected from the exit of the column at predetermined time intervals, and Cr(VI) concentration was determined using UV-Vis spectrophotometer (UV-1800 Shimadzu, Japan) at 540 nm after complex formation with 1,5 diphenyl carbazide.

The flow to the column was continued until the outlet concentration (C_t) approached the inlet concentration (C_0).

The breakthrough curves are obtained by plotting the dimensionless concentration C_t/C_0 versus time or volume of the effluent.

The total adsorbed Cr(VI) ions, q_t(mg/g), in the column for a specific flow rate and initial concentration is calculated using the following equation:

$$q = \frac{Q}{1000m}\left(C_0 t_s - \int_0^{t_s} C dt\right) \quad (1)$$

where C_0 and C (mg/L) are the initial and effluent concentrations at time t, respectively, q (mg/g) is the biosorption capacity of the bed, t (min) is the exhaustion time, Q (ml/min) is the flow rate, and m (g) is the mass of biosorbent.

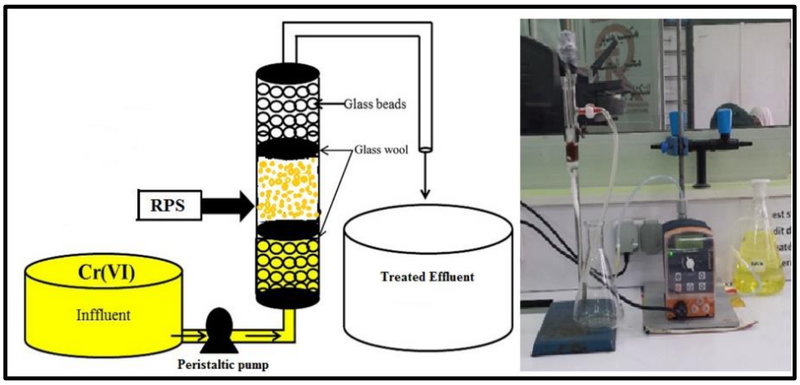

Fig.2. Schematic representation of fixed-bed column

3. RESULTS AND DISCUSSION

3.1. Characterization of the RPS biosorbent

Analysis by SEM

The scanning electron microscopy enables the direct examination in the surface microstructures of the raw peach stones biosorbent. The representative SEM images (Figure 3) show that the external surface of RPS presents irregular porous nature structure. This porosity was formed during the oven-heating step in the preparation of the adsorbent [23]. Such porosity represented a good possibility for the Cr(VI) ions to be adsorbed onto the RPS surface.

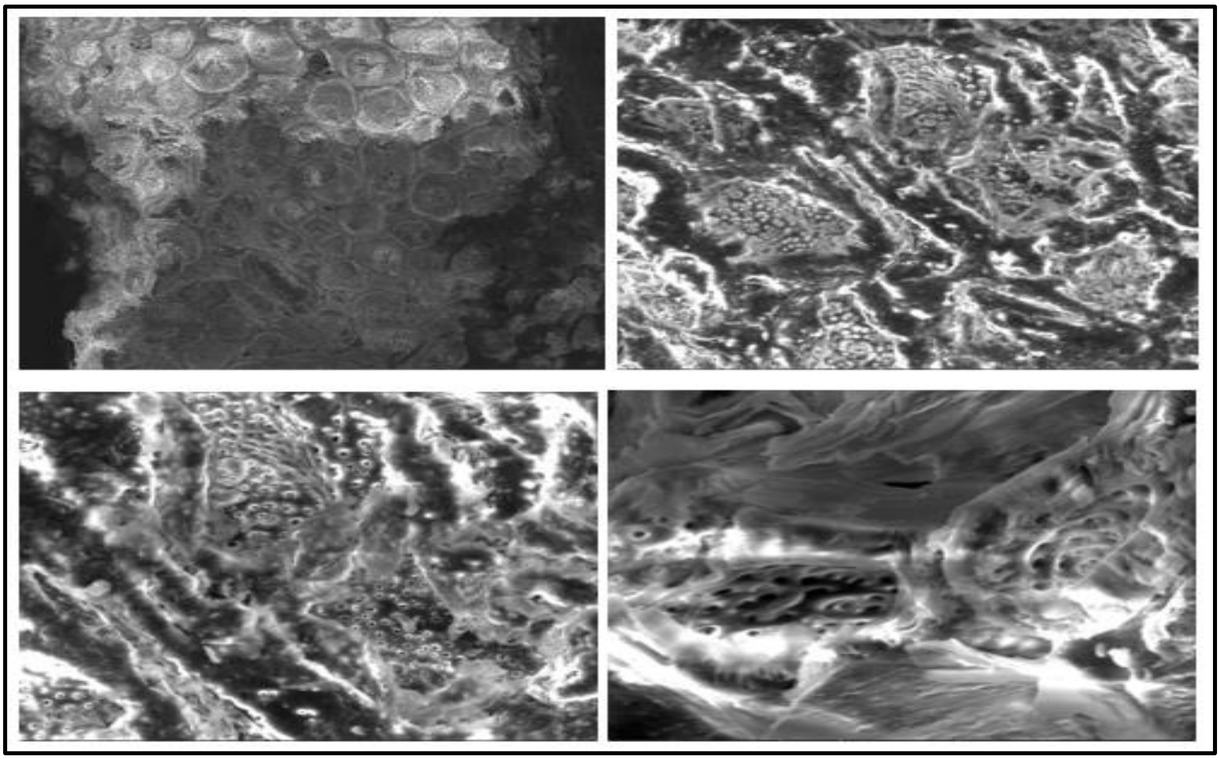

Fig. 3. SEM images of the raw peach stones adsorbent

FTIR analysis

The FTIR spectrum of the raw peach stones is illustrated in Figure 4 and the positions with assignments of bands are presented in Table 1. The main absorption bands were observed at 3440, 2925,1735,1633,1261 and 1047 cm^{-1}. The results indicated that there are a number of important functional groups on the surface of RPS.

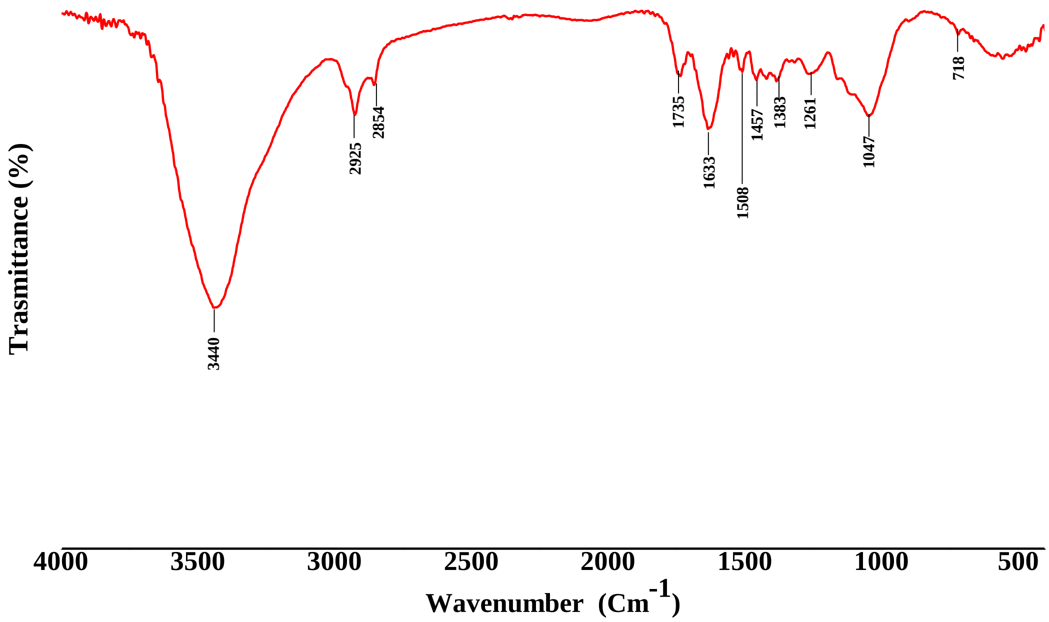

Fig. 4. FTIR spectra of raw peach stones (RPS)

Table 1. Assignment and positions of bands in the FTIR spectra of RPS biosorbent

Band position cm^{-1}	Assignment	References
3440	Carboxylic/OH stretching and N–H stretching	[1, 13]
2925	Aliphatic C–H stretching	[24, 25]
2854	–CH stretching vibration in –CH and $–CH_2$	[26, 27]
1735	C=O stretching vibration of carboxylic groups	[28, 29]
1633	C=C stretching vibration of aromatic ring structures	[24, 29, 30]
1508	C-C/ C=C stretching vibration of aromatic ring	[31]
1457	C-C/ C=C stretching vibration of aromatic ring	[31]
1420	C-C stretching of aromatic ring	[32]
1383	Symmetric bending of OCH_3 or $–CH_3$ symmetrical deformation mode (scissoring) in amide group	[13, 26]
1261	C–O stretching	[24, 33]
1047	C-C/ C=C stretching vibration of aromatic ring	[34, 35]
718	Torsional vibrations of benzene t(C-H)	[31]

3.2. Fixed bed column experiments

3.2.1 Effect of flow rate

Flow rate is an important parameter in evaluating adsorbents during continuous treatment of wastewaters on an industrial scale [36]. The effect of flow rate (Q in ml / min) on chromium (VI) adsorption by raw peach stones was studied at three different flow rates (1.5, 3 and 5 ml / min), while initial concentration and bed height were maintained at constant values of 30 mg /

L and 1 cm respectively. The breakthrough curves at different flow rates are illustrated in Figure 5. The results showed that the breakthrough time (t_b) decreased from 600 to 20 min with the increase in flow rate from 1.5 to 5 ml / min, this behavior can be attributed to insufficient residence time of the solute in the column, which caused the Cr (VI) solution to leave the column before equilibrium occurred. Insufficient contact time between the adsorbent and Cr (VI) at a higher rate leads to incomplete adsorption [37-39]. With increasing the flow rate from 1.5 to 5 ml / min, the saturation also occurs more rapidly, the exhaustion time (t_e) was reduced from 3000 to 660 min (Table 2). The adsorption capacity was decreased from 78.463 to 57.472 mg / g .When the flow rate is increased, the chromium (VI) ions do not have sufficient time to penetrate and diffuse deeply into the pores. Consequently, equilibrium does not take place, and lower absorption capacity might be achieved at higher flow rate [20, 40, 41]. In summary, lower flow rate allows for sufficient contact time between RPS (adsorbent) and Cr (VI (adsorbate) to reach their equilibrium, and the optimum flow rate was 1.5 ml / min. The results obtained are in accordance with previous studies announced in the literature reporting high adsorption of Cr (VI) at lower flow rates [42].

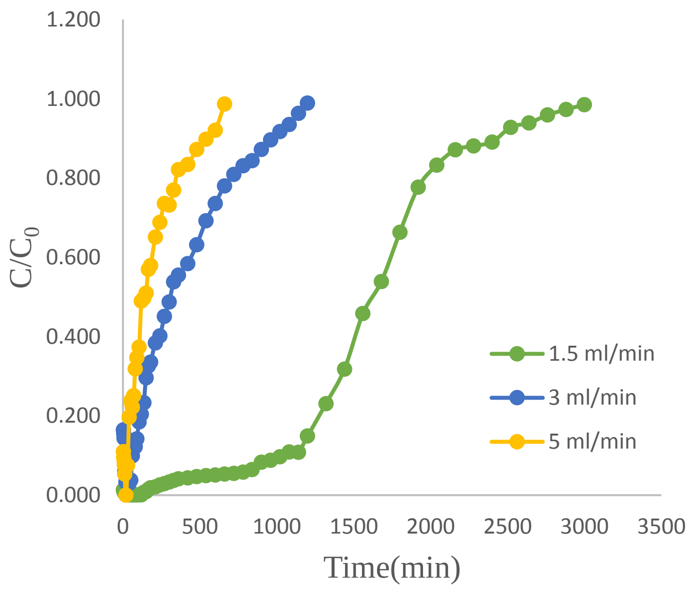

Fig. 5. Breakthrough curve for Cr(VI) biosorption onto RPS at different flow rates [Q (1.5, 3 and 5 ml/min), H (1cm), C_0 (30 mg/l), pH (2), T (25°C)]

3.2.2. Effect of bed height

The other factor that has the greatest effect on the absorption capacity of metals in a dynamic system is the height of adsorbent inside the column [20, 42]. The effect of bed height on chromium (VI) adsorption was studied at different bed heights (1, 2 and 3 cm), while the initial concentration and flow rate of the chromium solution maintained at 30 mg / L and 1.5 ml / min, respectively. The breakthrough curves for Cr(VI) adsorption obtained at different bed heights are shown in Figure 6. The results obtained show that the exhaustion time (t_e) increases from 3000 to 8880 min with the increase of the height of the bed from 1 to 3 cm (Table 2). With the increase in bed height, the breakthrough was delayed and column performance was improved (breakthrough time (t_b) increases from 600 to 1800 min). With increase in bed height, there is an increase in adsorbent amount thereby more surfaces available for adsorption and more contact time leading to higher adsorption of the metal [38]. Lower adsorption at lower bed height may be due to saturation in lesser time which leads to lesser breakthrough time [42]. The removal of metal ions usually depends on the amount of adsorbent available for adsorption [38, 39]. The values of the adsorption capacity of the column are 78.463, 89.802 and 96.791 mg / g for bed heights of 1, 2 and 3 cm, respectively (Table 2). The increase of adsorption capacity of metal with the increase in bed height might be due to the increase in the bed residence time and surface area of the adsorbent. Thus, more volume of effluent can be treated at longer bed height with a good efficiency of Cr (VI) removal. The result confirms that increase in column bed height increases the Cr (VI) removal efficiency of the RPS, and the optimum bed height for RPS in the column was 3 cm. Similar observations have been reported by Khitous et al. (2016) for Cd(II) adsorption by Pleurotus mutilus biomass in fixed bed column [43].

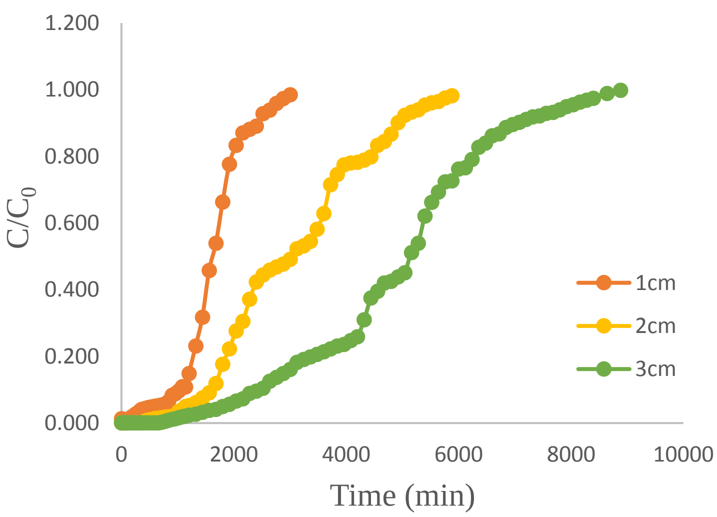

Fig. 6. Breakthrough curve for Cr(VI) biosorption onto RPS at different bed heights [H (1, 2 and 3cm), Q (1.5 ml/min), C_0 (30 mg/L), pH (2), T (25°C)]

Table 2. Experimental parameters obtained from breakthrough curves for Cr(VI) adsorption onto RPS

Q (ml/min)	H (cm)	t_b (min)	t_e (min)	q (mg/g)
1.5	1	600	3000	78.463
3	1	50	1200	62.739
5	1	20	660	57.472
1.5	1	600	3000	78.463
1.5	2	1140	5880	89.802
1.5	3	1800	8880	96.791

3.3. Breakthrough curve modeling

The Thomas and Yoon-Nelson models were proposed to illustrate column breakthrough curve obtained at different flow rates and bed heights.

Thomas model

The Thomas model is one of the most widely used models to describe the performance theory of the continuous adsorption process. This model was applied to the experimental data with respect to flow rate (Figure 7) and bed height (Figure 8) conditions. The parameters calculated from the linear representation of the model are summarized in Table 3. This model is expressed by:

$$\ln\left[\left(\frac{C_0}{C_t}\right)-1\right]=\frac{k_{Th}q_0m}{Q}-k_{th}C_0\,t \quad (2)$$

where k_{Th} is the Thomas model constant (ml/min g) , q_0 is the maximum adsorption capacity (mg/g), and t stands for total flow time (min).

The values of k_{Th} and q_0 can be determined from the linear plot of $\ln\left[\left(\frac{C_0}{C_t}\right)-1\right]$ against t.

As shown (Table 3), with increasing the bed height, the value of q_0 increased and the value of k_{Th} is decreased. In addition, the values of k_{Th} increases while those of q_0 decreases with an increase in the flow rate.

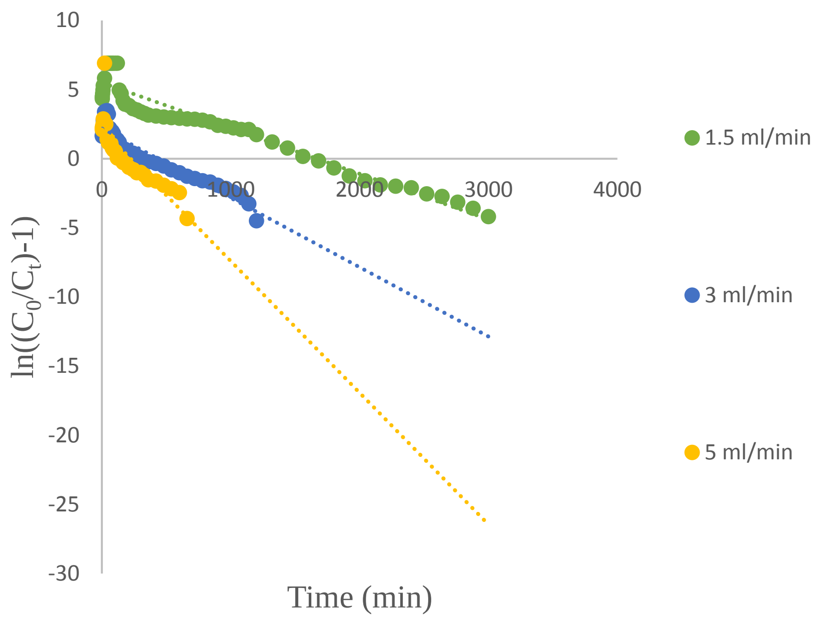

Fig. 7. Thomas model plot for Cr(VI) biosorption on RPS at different flow rates

[Q (1.5; 3 and 5 ml/min), H (1cm), C_0 (30 mg/l), pH (2), T (25°C)**]**

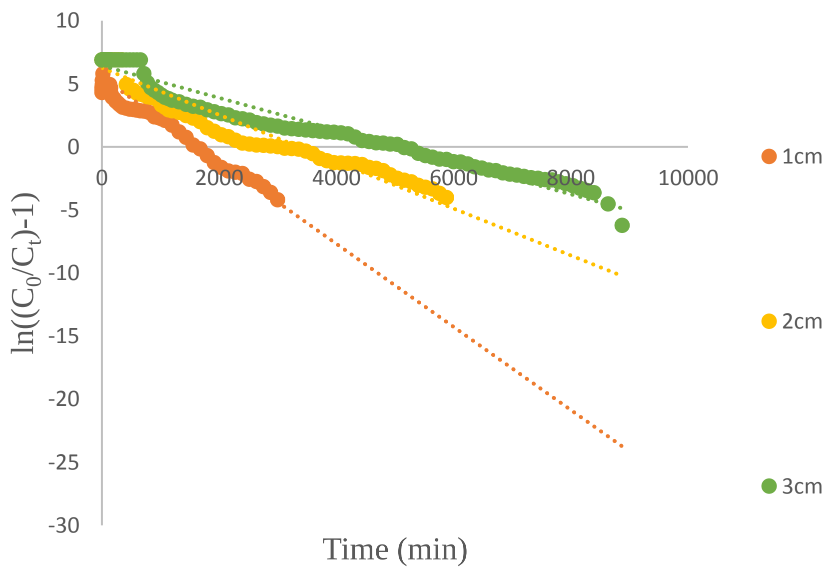

Fig. 8. Thomas model plot for Cr(VI) biosorption on RPS at different bed heights

[H (1, 2 and 3cm), Q (1.5 ml/min), C_0 (30 mg/L), pH (2), T (25°C)**]**

Yoon-Nelson model

Yoon-Nelson model was applied to the experimental data with respect to flow rate (Fig.9) and bed height (Fig.10) conditions.

The linear expression for this model is expressed as follows:

$$\ln\left(\frac{C_t}{C_0 - C_t}\right) = k_{YN}t - \tau k_{YN} \quad (3)$$

where k_{YN} is the rate constant (min^{-1}) and τ is the time required for 50% adsorbate breakthrough (min). A linear plot of $\ln\left(\frac{C_t}{C_0 - C_t}\right)$ against t is used to determine the values of k_{YN} and τ from the slop and intercept of the plot, respectively.

According to Table 3, as shown, increase in k_{YN} and decrease in τ was obtained by increasing the flow rate. On the other hand, when the height of the bed increases, k_{YN} decreases while the value of τ increases. A comparison of the R^2 values shows that both the Thomas and Yoon-Nelson models can be used to predict adsorption performance for column Cr (VI) adsorption.

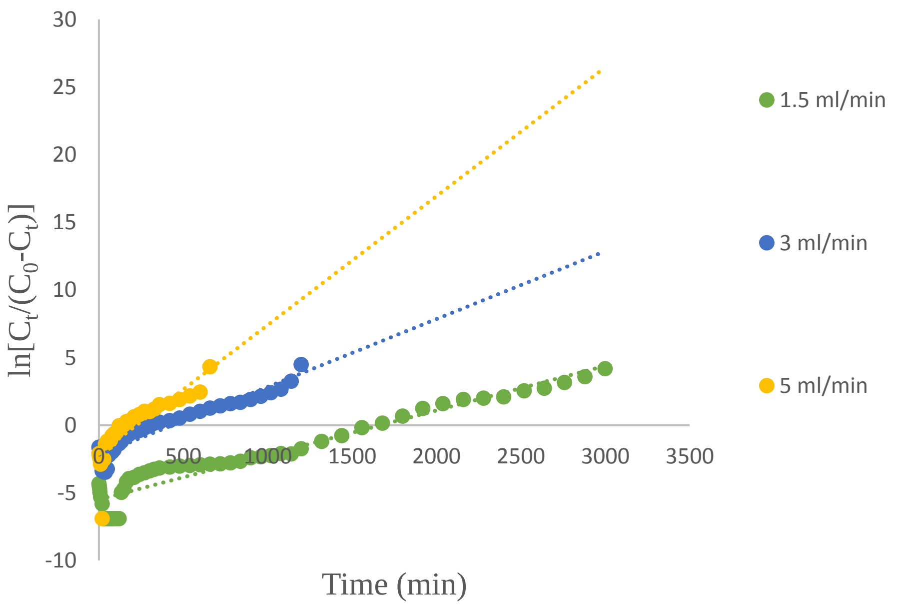

Fig. 9. Yoon-Nelson model plot for Cr(VI) biosorption on RPS at different flow rates. **[**Q (1.5; 3 and 5 ml/min), H (1cm), C_0 (30 mg/l), pH (2), T (25°C)**]**

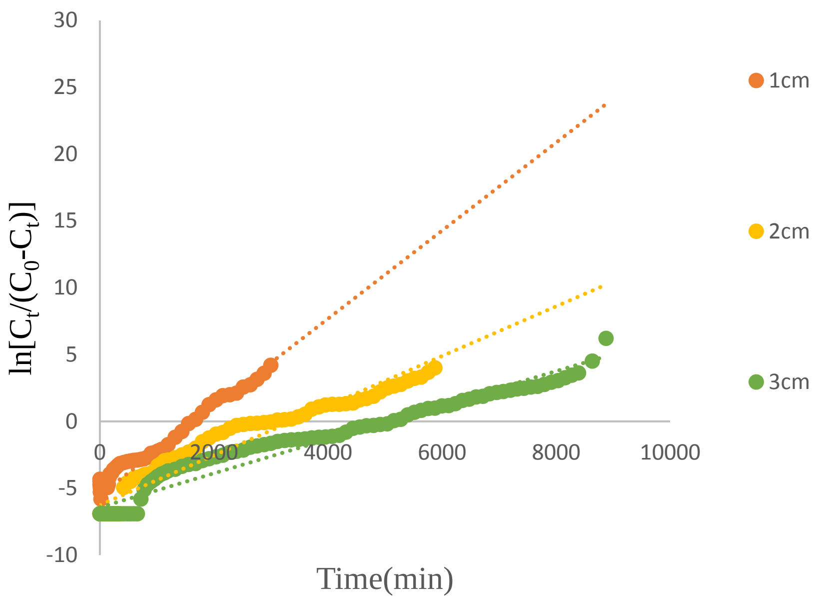

Fig. 10. Yoon-Nelson model plot for Cr(VI) biosorption on RPS at different bed heights. [H (1, 2 and 3cm), Q (1.5 ml/min), C_0 (30 mg/L), pH (2), T (25°C)]

Table 3. Thomas and Yoon-Nelson models' parameters for Cr(VI) adsorption onto RPS at different flow rates and bed heights

Flow rate (ml/min)	Bed height (cm)	Thomas model			Yoon-Nelson model		
		k_{th} (ml/min.g) * 10^{-4}	q_0 (mg/g)	R^2	k_{YN} (min^{-1})	τ (min)	R^2
1.5	1	1.100	43.555	0.9206	0.0033	1664.788	0.9206
3	1	1.666	22.826	0.9029	0.0050	436.060	0.9029
5	1	3.166	19.484	0.732	0.0095	223.368	0.732
1.5	1	1.100	43.555	0,9206	0.0033	1664.788	0.9206
1.5	2	0.633	50.328	0.9401	0.0019	3293.053	0.9401
1.5	3	0.433	53.980	0.9602	0.0013	4948.000	0.9602

4. CONCLUSION

The raw peach stones biomass was found to be an effective low-cost biosorbent for the removal of toxic hexavalent chromium from aqueous solution.

Based on the experimental results, the following conclusions can be listed:

- The FTIR analyses showed presence of significantly functional groups on the RPS surface.

- The results indicate metal uptake capacity to be strongly affected by parameters such as bed height and flow rate.
- The bed height of 3cm and low flow rate of 1.5 ml/min are the optimum conditions, and the maximum capacity of RPS in column investigation was found to be about 96 mg/g under the selected operational conditions (Flow rate=1.5ml/min, bed height=3cm C=30mg/l and pH=2)
- Mathematically, breakthrough data presented a good match with both the Thomas and Yoon-Nelson models
- Utilization of the biomaterial with no treatment needed can be considered as environmentally friendly and lies in the green analytical chemistry rules: reduction in the use of reagents and energy, agricultural waste minimization.

Further, it can be concluded that RPS is a good potential, inexpensive and eco-friendly biosorbent for the removal of Cr(VI) from aqueous solution in fixed-bed column.

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